STANDARD OPERATING PROCEDURE

for

SOLID MATERIAL VOLATILE ORGANIC COMPOUND SAMPLING FOR TOTALS ANALYSIS

Revision 02

Remediation Services Division, OEA
Surveillance Division, OEC
Laboratory Services Division, OEA

Office of Environmental Assessment
Office of Environmental Compliance

Louisiana Department of Environmental Quality

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Document Review and Revision Record

Note: Actions older than 5 yrs may be removed from this record

Date	Revision No.	Record of Activity
6/28/02	0	Initial document approved.
3/13/03	1	Revise Cover Page; Add Document Review Record; Update Table of Contents; Update Formatting throughout document; Section 1 - Link to EPA clarification memo also updated; Section 3.1 thru 3.3 - Text was edited to add Terracore equivalent samplers; Section 3.5 - Document edited to remove limitations on the Encore^{tm} samplers use for non-cohesive materials; Added Section 4 – Quality Assurance and Quality Control
8/18/05	2	Update to the new SOP template; added Definitions /Acronyms/Abbreviations, Personnel Qualifications, Records Management, SOP Review/ Revision/ Approvals, References, and Attachment Sections; updated administrators and assistant secretary names on approval page; updated LSD to Office of Environmental Assessment; minor wording changes; added reference to FAQ web posting.

Table of Contents

1.0 Purpose / Applicability

1.1 The purpose of this SOP is to provide procedures for LDEQ personnel to employ Method 5035 when taking samples of solid materials for total volatiles analysis during their duties. Method 5035 is to be used exclusively for solid material sampling on and after July 1, 2002.

1.2 In June 1997 the United States Environmental Protection Agency (EPA) adopted Method 5035 for the sampling and analysis of total amounts of volatile organic compounds (VOCs) in solids. Solids may include soil, sediment, tank bottoms, filter media, and other solid materials. This method was adopted in the Third Edition revisions of SW-846. This method does not apply to samples collected for leachate analysis such as those for the Toxicity Characteristic Leachate Procedure (TCLP), or the Synthetic Precipitation Leaching Procedure (SPLP).

1.3 Method 5035 was adopted because of studies showing that sampling according to the previous methods resulted in significant losses of selected VOCs. Method 5035 incorporates chemical preservatives and sample storage techniques to limit volatilization and biodegradation of VOCs. SW-846, which contains Method 5035, can be found on the Internet at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm, and an EPA clarification of the method can be found at http://clu-in.org/goto.cfm?link=%2Fproducts%2Fregs%2Fsw846mem%2Ehtm&id=75.

1.4 Solid material sampling is performed by a variety of LDEQ personnel in various divisions to provide quantitative analytical data for total amounts of specified constituents of concern (COC) in the media sampled. This analytical data is then compared to regulatory levels, Risk Evaluation/Corrective Action Program (RECAP) Standards, or other benchmarks to provide information for decision-making. The goal of such sampling is to provide the most accurate information possible on the total concentration of specified COC in the solid media sampled.

2.0 Definitions/Acronyms/Abbreviations

2.1	LDEQ	Louisiana Department of Environmental Quality
2.2	EPA	Environmental Protection Agency
2.3	VOC	Volatile Organic Compounds
2.4	TCLP	Toxicity Characteristics Leachate Procedure
2.5	COC	Constituents of Concern
2.6	SPLP	Synthetic Precipitation Leaching Procedure

2.7 DQO Data Quality Objectives

2.8 MS/MSD Matrix Spike / Matrix Spike Duplicate

2.9 RECAP Risk Evaluation Corrective Action Program

2.10 FID Flame Ionization Detector

2.11 PID Photo-ionization Detector

2.12 DOT Department of Transportation

2.13 LSD Laboratory Services Division

2.14 QA/QC Quality Assurance / Quality Control

2.15 QAR Quality Assurance Representative

3.0 Personnel Qualifications

3.1 Personnel from RSD, SD, and LSD by the requirements of their job descriptions and through ongoing training are qualified to perform the task required by this procedure.

4.0 Procedure

4.1 Pre-sampling Requirements

4.1.1 Identify and justify the numbers and locations of solid samples to be taken. This should be based on EPA SW-846 Chapter 9, DQOs, best professional judgment, or LDEQ policy. Solid materials include soil, sediment, and solid wastes that are solids or semi-solids.

4.1.2 Determine the Method 5035 option to be used to collect the samples. This is a choice between LDEQ approved airtight coring devices, or preserved vials. This determination should be based on holding time, laboratory-processing considerations, soil type (calcareous soils have special considerations when using the preserved vial option), shipping considerations, etc.

4.1.3 Determine the number of volatile sample containers required for the sampling episode. NOTE: Extra containers may be needed for field testing, rejection of weights, or other problems with sample collection (see attached Example Sample Container Requirements table).

4.1.4 Request sample containers using the LDEQ Sampling Request Form, available from the LDEQ-LSD. Requests for containers should be made when possible at least one week prior to the sampling event. Otherwise notification should be given as soon as the decision has been made to collect VOC samples. The LDEQ-LSD will make final recommendations on numbers of additional sample containers required based on the analytical methods to be employed following sample collection, the need for MS/MSD sample(s), etc.

4.1.5 A determination of whether the sample will be considered high (>200 µg/Kg) or low (0.5-200 µg/Kg) concentration should be performed if the preserved vial option is to be employed. This may be based on DQOs, expected concentrations, regulatory limits, or RECAP Standards that are used as a basis for comparison, etc. This can be accomplished by headspace analysis using a portable instrument such as a FID, PID, or prior analytical data. NOTE: If the expected concentrations cannot be estimated prior to sampling, and/or DQOs or other considerations indicate the need for both concentration levels, both low and high concentration aliquots must be collected.

4.1.6 The method and this SOP call for the use of a portable balance if the preserved vial option is to be employed. Prior to the sampling event, sampling personnel must become familiar with the operation of the portable balance(s) available. The balance used must be calibrated and operated in accordance with the manufacturer's instructions. Balances used in the field should be checked against an appropriate reference weight at least once daily, prior to weighing samples, or as described in the sampling plan. Calibration weights and fresh batteries should also be taken to the field during sampling in case battery replacement and/or re-calibration are needed during the sampling event. The balance shall be operated in a wind-protected environment. Sampling personnel should plan to use tents, tarps, an enclosed vehicle, or other protection to ensure accurate weights are obtained.

4.1.7 Non-cohesive solids and solid wastes that will not readily form a cohesive plug may not be amenable to collection with plastic syringes or the Terracore sampler (or equivalent) if the preserved vial option is to be employed, or an airtight coring device such as the Encore sampler. These aliquots may have to be collected using a spatula or scoop. The aliquot should be handled as little as possible during the sampling event to avoid loss of volatiles. The spatula or scoop must have been properly cleaned and included in the equipment rinseate QA/QC sample to confirm cleanliness. Terracore samplers (or equivalent) must be stored in a clean sealed container. This container should not be opened in a potentially contaminated area.

4.1.8 If using the preserved vial option and low concentration aliquots are to be collected, check for solid reactivity (effervescence) by placing an aliquot of the solid into a vial containing sodium bisulfate solution. If the solid reacts, do not

use the sodium bisulfate preservative for low concentration samples. Use either organic-free reagent water or an airtight coring device.

4.1.9 Sampling personnel should be aware that U.S. Department of Transportation (DOT) rules and regulations for safe transport of dangerous goods and hazardous materials, Code of Federal Regulations, Title 49 Parts 173-180 (49 CFR 173-180), may apply to certain samples. These regulations may apply to methanol and sodium bisulfate preserved samples if a commercial carrier such as FedEx transports them to the laboratory. Persons shipping such samples may be required to receive training and refresher training in these regulations. Specific packaging, labeling, and documentation are required by DOT regulations for such materials. Further information on shipping considerations for the preserved vial option can be found on the Internet at http://hq.environmental.usace.army.mil/tool_info/lessons/list/ll47/ll47.html

4.1.10 Sampling personnel should use disposable vinyl or latex gloves during sampling to avoid direct contact with potentially contaminated media and preservatives. If using the preserved vial option, safety glasses or goggles should be worn during sampling due to the possibility of splashing preservatives into the eyes when extruding sample aliquots into the preserved vials. Site-specific conditions may necessitate the use of additional personal protective equipment.

4.1.11 The use of a trip blank is required when performing sampling for volatiles. Trip blanks should be supplied by the LDEQ-LSD, and kept in each sample cooler that is to be used for the volatile sample aliquots. It is then returned along with the samples for analysis. This is one of the QA/QC samples required for routine sampling events. See Section 2.4 of the RECAP document for further information on required QA/QC samples.

4.2 Preserved Vial Option Sample Collection

4.2.1 General Procedure for all sample collections

4.2.1.1 Prior to adding solid to any vial, the individual vial weight must be checked using a portable balance (unless the vials were weighed or weight checked in the laboratory 24 hours before sample collection). If the weight obtained on the portable balance in the field differs from the laboratory weight (marked on the vial) by +/- 0.2 g, the vial must not be used; return it to the laboratory for proper disposal. This vial weight check is to be performed no more than 24 hours prior to sample collection. This step is necessary to insure the vials have the correct volume of preservative, organic free water, or methanol.

4.2.1.2 Using a disposable Terracore sampler (or equivalent sampler), collect 5 g (+/- 0.5 g) of the solid to be analyzed as soon as possible after the surface of the solid has been exposed to the atmosphere.

4.2.2 Collection of non-calcareous or non-reactive solid low concentration samples (0.5 – 200 µg/Kg)

4.2.2.1 Place the solid in a laboratory-supplied vial containing sodium bisulfate solution and a magnetic stir bar. It is advised to place the preserved vial on the field balance and tare the balance so the vial and preservative weight is zeroed out, then the solid can be added till 4.5-5.5 g is placed in the container. If 5.5 g is exceeded, do not use the container for analysis; return it to the laboratory for proper disposal. Alternately, the Terracore sampler (or equivalent sampler) can be placed on the field balance and its weight tared out. Then it can be used to collect the sample aliquot. The sampler and aliquot can then be placed on the field balance and checked to insure the aliquot is 4.5-5.5 g. This aliquot can then be extruded into the vial.

4.2.2.2 Go to step 4.2.5 to complete sample collection process.

4.2.3 Collection of calcareous or reactive solid low concentration samples (0.5 - 200 µg/Kg)

4.2.3.1 Place the solid in laboratory supplied vial containing organic-free reagent water. It is advised to place the vial on the field balance and tare the balance so the vial and water weight is zeroed out, then the solid can be added till 4.5-5.5 g is placed in the container. If 5.5 g is exceeded, do not use the container for analysis. Alternately, the Terracore sampler (or equivalent sampler) can be placed on the field balance and its weight tared out. Then it can be used to collect the sample aliquot. The sampler and aliquot can then be placed on the field balance and checked to insure the aliquot is 4.5-5.5 g. This aliquot can then be extruded into the vial.

4.2.3.2 Go to step 4.2.5 to complete sample collection process.

4.2.4 Collection of high concentration samples (> 200 µg/Kg)

4.2.4.1 Place the solid in the weight-checked laboratory-supplied vial containing 5-mL methanol. It is advised to place the vial on the field balance and tare the balance so the vial and methanol weight is zeroed out, then the solid can be added till 4.5-5.5 g is placed in the container. If 5.5 g is exceeded, do not use the container for analysis; return it to the laboratory for proper disposal. Alternately, the Terracore sampler (or equivalent sampler, or cut-off syringe) can be placed on the field balance and its weight tared out. Then it can be used to collect the sample aliquot. The sampler and aliquot can then be placed on the field balance and checked to insure the aliquot is 4.5-5.5 g.

This aliquot can then be extruded into the vial.

4.2.4.2 Go to step 4.2.5 to complete sample collection process.

4.2.5 Complete sampling collection process

4.2.5.1 Quickly brush any solid off the vial's sealing surfaces and threads to ensure an airtight seal. Immediately seal the vial.

4.2.5.2 Record the weight of the vial, preservative, and added solid, to the nearest tenth of a gram on the vial with an indelible marker capable of writing on glassware (such as a Sharpie).

4.2.5.3 Also collect a bulk aliquot container following procedure in section 4.3 of this SOP.

4.2.5.4 Place the containers in a plastic bag and seal.

4.2.5.5 Store samples on ice at approximately 4° C and deliver to laboratory. If samples were collected in water, place vials on dry ice at an angle to prevent the water from shattering the vial due to expansion during freezing.

4.3 Collection of Bulk Aliquot

4.3.1 Using an appropriate collection device, place enough solid in a 4 ounce wide-mouth jar to eliminate any headspace. Quickly brush any solid off the sealing surfaces and threads and seal immediately. This aliquot will be used for laboratory screening.

4.3.2 Store on ice at approximately 4° C and deliver to laboratory.

4.4 LDEQ Approved Airtight Coring Device Option

4.4.1 Airtight coring devices, such as the EnCore™ Sampler, may be used in place of vials. However, this option requires close coordination with the laboratory. This type of device requires laboratory processing of the aliquots within 48 hours of collection therefore the aliquots should be delivered to the laboratory as soon as possible after collection. The laboratory must be informed when using these devices to ensure that they have the ability to handle the samples appropriately and in a timely manner.

4.4.2 These devices must be used in accordance with the manufacturers' instructions. Sampling personnel must read and become familiar with these instructions prior to use of such a device.

4.4.3 Collect a bulk aliquot when using an airtight coring device following section 4.3 of this SOP.

5.0 Records Management

5.1 All data / information collected, as described within, as described within this SOP, must be documented in permanent files.

5.2 All-important changes that have a bearing on subsequent reviews will be noted in the "Document Review and Revision Record" and retained for a period of one year by QAR.

5.3 As various Sections, Divisions, or Offices within LDEQ may require this document, the latest version is to be accessible via the LDEQ intranet. Refer to the SOP database to verify the latest revision is being utilized.

6.0 Quality Assurance and Quality Control

6.1 All required data and information is to be captured on the LSD chain of custody form. DQOs should be identified in advance to plan for the appropriate number and type of aliquots.

6.2 The preserved vial option requires that a field weight check be performed to ensure the appropriate amount of soil and preservative are obtained. Personnel should be familiar with the manufacturers instructions for the use and calibration of all equipment, particularly the field balance used for weight checks. Calibration should only be performed utilizing certified calibration equipment and weights.

6.3 The airtight coring device option requires coordination with the Laboratory to ensure that the 48-hour time to extraction is not exceeded. If this time limit cannot be met, the preserved vial option must be utilized.

6.4 The TL, as the point of contact for all issues relating to a site, is responsible for the oversight of the sampling process, and any other actions necessary to ensure the successful completion of Solid Material VOC Sampling as reflected in this document.

7.0 SOP Review/ Revision/ Approval

7.1 One or more individuals with appropriate training and experience with the process should review the SOP. This is normally the Development Team and Lead Developer.

7.2 The finalized SOP should be approved as described in the organizations Quality Management Plan. Generally the Lead Developer, the Administrator(s), and

possibly an Assistant Secretary review and approve each SOP by signature, which indicates that the SOP has been reviewed by management.

7.3 The SOP needs to remain current. Therefore, the SOP is to be reviewed on a periodic basis to ensure the policies and procedures remain current and appropriate, or to determine whether the SOP is still needed.

8.0 References

8.1 SW-846 Method 5035, Closed-system purge-and-trap and extractions for Volatile Organic in soil and waste samples
http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm

8.2 EPA's clarification of SW-846 Method 5035
http://clu-in.org/goto.cfm?link=%2Fproducts%2Fregs%2Fsw846mem%2Ehtm&id=75.

8.3 US Department of Transportation (DOT) –Transportation of dangerous goods and hazardous materials – 49 Code of Federal Regulations 173-180

8.4 Shipment of Methanol or Sodium Bisulfate Preserved Environmental Samples
http://hq.environmental.usace.army.mil/tool_info/lessons/list/ll47/ll47.html

8.5 VOC sampling Frequently Asked Questions
http://deq.louisiana.gov/laboratory/method5035FAQ.pdf

9.0 Attachments

	Title
Appendix A	Sample container requirements table

Appendix A
SAMPLE CONTAINER REQUIREMENTS[1]
NOTE: Also collect a 4 oz bulk aliquot for each sample, see Section 3.4

Concentration of / Solid Volatiles Type	<200µg/Kg	>200µg/Kg	Unknown Concentration
Non-reactive	3 - 40mL vials with 5mL of organic-free reagent water, 1 g $NaHSO_4$ and a magnetic stirring bar weighed to the nearest 0.01g[3].	3 - 40mL vials with 5mL of methanol weight checked to the nearest 0.01g[3].	3 - 40mL vials with 5mL of organic-free reagent water, 1 g $NaHSO_4$ and a magnetic stirring bar weighed to the nearest 0.01g[3]. **And** 3 - 40mL vials with 5mL of methanol weighed to the nearest 0.01g[3].
Reactive	3 - 40mL vials with 5mL of organic-free reagent water weighed to the nearest 0.01g[3]. OR 3 EnCore samplers[2].	3 - 40mL vials with 5mL of methanol weighed to the nearest 0.01g[3].	3 - 40mL vials with 5mL of organic-free reagent water weighed to the nearest 0.01g[3]. OR 3 EnCore samplers[2]. **And** 3 - 40mL vials with 5mL of methanol weighed to the nearest 0.01g[3].
Unknown	3 - 40mL vials with 5mL of organic-free reagent water, 1 g $NaHSO_4$ and a magnetic stirring bar weighed to the nearest 0.01g[3]. AND 3 - 40mL vials with 5mL of organic-free reagent water weighed to the nearest 0.01g[3]. OR 3 EnCore samplers[2].	3 - 40mL vials with 5mL of methanol weighed to the nearest 0.01g[3].	3 - 40mL vials with 5mL of organic-free reagent water, 1 g $NaHSO_4$ and a magnetic stirring bar weighed to the nearest 0.01g[3]. **And** 3 - 40mL vials with 5mL of organic-free reagent water weighed to the nearest 0.01g[3]. OR 3 EnCore samplers[2]. **And** 3 - 40mL vials with 5mL of methanol weighed to the nearest 0.01g[3].

[1] - Supplied by the laboratory

[2] - Or other equivalent LDEQ approved airtight coring device. Notify the laboratory of your sample needs. If airtight samplers are to be used, coordinate with the laboratory to ensure that the aliquot(s) can be processed within 48 hours.

[3] - The vials will be pre-weighed by the laboratory to the nearest 0.01g. If the required weight check is performed in the field, a variance of up to 0.2 g is allowed. If the required weight check is performed in the laboratory, a variance of up to 0.01g is allowed. Weight checks must be performed within 24 hours of use.